Strategy for using real-time XRF and incremental-composite sampling to delineate Pb-contaminated area for soil removal

Strategy summary: Real-time XRF and composite sampling are used to iteratively zero in on the footprint of soil to be removed. Data uncertainty (due to soil heterogeneity) and decision uncertainty (due to data gaps) are identified and resolved in real-time, guaranteeing a successful cleanup of the Pb area. There is no chance that a second mobilization could be triggered by unexpected exceedances when cleanup confirmation samples come back from the lab weeks to months after the field work.

Before field mobilization

#B1: Work with risk assessor(s?), the state(?), and the RPM to determine threshold concentration for soil requiring removal due to elevated Pb.

- Determine if the local background concentration (outside of the refinery area) is a consideration for setting the threshold, or are they only concerned about potential risk from on-site exposure or from off-site migration of contaminated soil.
- In other words, will the default of 400 ppm Pb be used, or another number (either higher or lower)?
- Determine the depth component:
 - o To what depth will surface soil be tested?
 - o Will testing of 1 or more subsurface depth intervals be required?

#B2: Determine removal options (backhoe & size of backhoe? hand-dig with shovel? other?), and disposal costs for removed soil.

- This information will help determine
 - o how "fine-grained" or "surgical" the Pb removal footprint should be,
 - o which determines how many iterations of removal boundary refinement (Steps #D5 and 6) are required.
- Will disposal option and cost depend on how high the soil Pb concentration is?
 - o This seems unlikely, but given the extremely high concentrations present, it needs to be checked.
 - o If there is a substantial differential cost for higher Pb concentrations, more sampling might be required to identify that soil, and
 - o Plans and equipment needed to keep soils of different concentrations segregated.
- What is the minimum amount of soil to be removed for a backhoe (or other rented machinery) to be cost-effective? Based on previous XRF work, might that minimum be exceeded?

#B3: Determine whether the RPM, risk assessor(s) or state staff wants ICP Pb data as verification of complete and effective cleanup.

- Determine number of ICP results they'd like to see (based on XRF concentration? or area?) in order to estimate lab costs
- Develop statistically sound strategy for the number of cleanup verification ICP samples and their selection if requester(s) unsure how many desired
 - o Maybe 1 DU and triplicate ISM samples over entire cleanup area to get 95%UCL?
 - o Might more than 1 DU over removal area be needed?

o Should ICP cleanup verification sampling be done immediately post-excavation, or after restoration back to grade? Or both?

#B4: Set-up XRF QC worksheets and sample concentration calculators

- Perform advance QC on XRF instrument to be used (if necessary because it was not done
 with that instrument before; will not be necessary if the TIIB XRF is used), select SRMs
 to use as LCSs and set up the LCS QC control charts.
- Determine the acquisition times to be used for LCSs and samples (if times are not the same)
 - o the samples can probably be run at 15 secs per XRF reading.
- Also set up 3 versions of the project's automatic XRF statistical worksheet for sample bag concentration determination.
 - o Determine data quality goals (width of confidence intervals or limit on %RSDs) for initial, delineation, and cleanup confirmation data sets
 - O As part of the statistical sample concentration worksheet for the Initial Data Sets (from Steo#D1), set up automatic %RSD calculations using the final bag average concentrations from the following SUs groupings
 - for each duplicate SU pair,
 - for all 8 SU samples as a group,
 - for the 4 SU areas to the left,
 - for the 4 SU areas to the right, and
 - for the 4 duplicate SU averages.
 - Determine expected differences between grouping %RSDs to confirm the effectiveness of the SU area and number of increments

#B5: On ERT's Pb data map, see if there is data that indicates where the Pb concentration seems to be consistently below the threshold concentration selected in #B1.

- Draw a line at this border to propose where the "clean" area possibly/probably begins.
- Also circumscribe a line around a proposed edge of the "hot" removal area (as currently understood from prior XRF data and the threshold established in #B1).
- If the decision in Step #B2 is that higher Pb concentrations will be segregated, a border for this area must also be proposed.

#B6: Gather supplies:

- QC'd thin-walled plastic read bags and thick-walled plastic sample handling and storage bags
- 10- & 60-mesh sieves
- XRF set-up in stand (if amenable to real-time analysis in the field
 - o use the XRF in handheld-mode if a stand is not feasible,
 - o charged batteries (as needed), and
 - o charged laptop (as needed)
 - o determine the most convenient electrical power options for charging batteries
- pogo-sampler and/or soil scoops and/or soil corer samplers (as needed based on selections in #B1 and #B2);
- rubber mallet, butcher paper, cookie trays, and Al foil
- PPE: gloves, dust masks, eye protection
- labeling supplies, scotch tape
- sample log sheets or log book

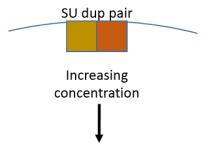
#B7: Plan field mobilization for weather such that field soil will be relatively dry.

- The rest of this outline assumes field soils (including at depth, if applicable) are not too wet to sieve.
- If it is possible that field work may be conducted when soils are too moist, a contingency plan for this possibility will also need to be developed.

During field mobilization

#D1a. INITIAL DATA SET: Start at the proposed edge of the removal area (as determined in #B5) and at the 4 cardinal compass points around the circumscribed area.

• At each of the 4 locations, mark off 2 adjacent 2x2 sq.ft. (may be scaled down to 1x1 sq.ft. or up to 3x3 sq.ft. or other as indicated) SU areas such that the bases of the SUs are set on the inside of the circumscribing line (as illustrated).



- Using the appropriate sample collection tools, take a composite sample having no less than 9 increments (3 rows of 3) per SU.
 - o Take the surface SU increments to the depth set in #B1.
 - o If Step #B1 determined that subsurface depth interval(s) are to be done, collect subsurface and surface depth intervals from sufficiently long cores.
- A total of 4 adjacent pairs of SU samples will be collected along the "hot" edge. Each adjacent pair is a duplicate set, for a total of 4 duplicate sets, 1 set at each cardinal compass point.

#D1b. Disaggregate the soil samples using the most efficient mechanisms for that soil. Use the 10-mesh sieve (if needed) to remove over-sized or extraneous materials (or hand-pick if very few). Place the <10-mesh portion in a "read-bag."

• Begin XRF readings with 3 shots on each side of the bag. Enter data in the calculation spreadsheet. If needed to attain statistical data quality goals, take additional shots on the bag (in sets of 2: one on each side of the bag for each iteration) until data quality goals are met.

#D2. Repeat Steps #D1a and D1b for the Initial Data Sets on the proposed edge of the clean area.

#D3. Evaluate the performance of the 2x2 (or other) sq.ft. SU composite area by comparing the %RSDs for the paired SUs against the %RSDs for the 4 cardinal directions

• If the paired SU %RSD is too high relative to the locations' %RSD, consider decreasing or increasing the SU area and/or increasing the number of increments per SU

#D4. Evaluate whether the 4 SU pairs on the "clean" line all had concentrations that were "clean" according to the threshold value chosen in #B1.

• If not, the "clean" line in that area will need to be moved further away and another pair of SUs evaluated at the new line.

#D5. DELINEATION DATA SETS: Using professional judgment and prior XRF data, circumscribe a line about midway between the "clean" and "hot" lines.

- Place a single SU (of the final selected area and number of increments) at the 4 cardinal directions AND at the 4 primary inter-cardinal directions (for a total of 8 individual SUs, each centered on 1 of 8 equal-length line segments).
- Process and analyze the bagged samples; use the bag mean as the sample concentration

#D6a. Move the "clean" and removal line segments as indicated by the concentrations of the 8 SU samples of #D5. High concentrations move the line segment outward; clean concentrations move the line segment inward.

#D6b. Collect another 8 2x2 sq.ft. SU samples. Use professional judgment to place the new round of 8 SUs in relation to it corresponding line segment (centered again or offset).

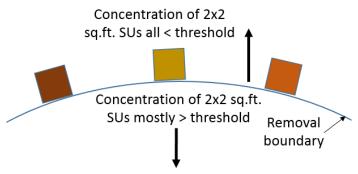
#D6c. Repeat #D6a to b as necessary to achieve the desired degree of "surgical" removal determined in #B2. Take into account any subsurface depth intervals being collected.

#D6d. Final boundary decisions should be based on the 95% UCL on the SU bagged samples, rather than the mean used before.

#D7a. When the desired degree of confidence in the clean/hot boundary is attained, determine the most efficient excavation footprint and draw the removal boundary (also at depth as indicated). If the team has high confidence in all parts of the removal boundary drawn in #D7a, proceed to excavation. If confidence is lacking, continue with #D7b.

#D7b. OPTIONAL: If desired, the removal boundary (or parts of the removal boundary) can be confirmed with a line of "confirmation" SUs having their edge placed to confirm "clean" soil beyond the boundary.

- Offset the confirmation SUs to "fill in" obvious spatial gaps along the boundary.
- The density of SUs along the boundary should provide a level of certainty commensurate with satisfying the balance among disposal cost vs. risk vs. the cost/time of sampling additional SUs.



• If the soil sampled so far has contained an even distribution of particle sizes and if particle effects were observed during sample analysis, it might be useful to put the

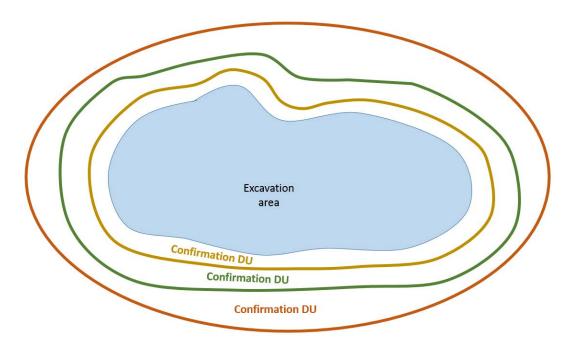
cleanup confirmation SU samples through an additional processing step prior to XRF analysis.

- O Particle effects observed during XRF analysis may indicate a marked difference in Pb loadings on smaller vs. larger soil particles. Since smaller particles fractions are more mobile by wind and runoff (and pose a greater exposure risk after transport) than larger particle fractions, determining the Pb concentration of the "fines," and adjusting the removal boundary accordingly, may be desirable.
- o Sieve the disaggregated sample through a 60-mesh sieve to isolate the <250-micron particle size. This fraction is placed in an XRF read-bag and analyzed with XRF. The 95% UCLs for the SU bags must be less than the decision threshold selected in #B1. If any are not, the boundary must be moved further out.

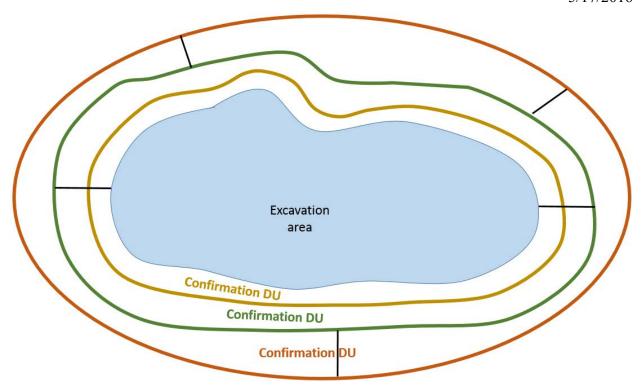
#D8. After soil removal is completed, the excavation (or post-restoration area?) will be designated as a DU(s) and sampled using at least 30 increments and with triplicate field samples to calculate a 95% UCL for the excavation area.

#D9. OPTIONAL: If desired for cleanup confirmation purposes around the excavated area before rented equipment is taken off-site, concentric bands surrounding the excavation can be marked off using string and/or flags.

- Triplicate incremental samples should be collected from the band DU nearest the excavation.
- If the first band is "clean," replicate incremental samples are not required for any outer bands.
- If the first band exceeds the threshold, the entire band may be excavated, or
- If more surgical removal is desired, several 2x2 sq.ft. SUs may be placed around the band to determine if only a small area is the problem.



• If the initial areas of the confirmation bands are large, the bands can be broken into smaller DUs, as illustrated below.



#D10. If ICP Pb data is desired for cleanup verification (as determined in #B3), and if it is acceptable to use soil from the XRF'd cleanup verification sample bags,

- Use incremental subsampling to remove 2 grams into a read bag (a smaller size, or a heat sealer may be needed to reduce the bag's volume).
- Record the XRF readings and statistics on the bag.
- Send the bagged 2-gram subsample for ICP analysis.
- Since heterogeneity-controlled ICP samples give Pb results that are the same (or much more commonly, less than) the XRF Pb results, the ICP cleanup verification samples are highly likely to come back "clean" as expected.

Prepared by Deana Crumbling, 17May16 For the Wilcox Superfund Site